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PATENT ABSTRACTS OF JAPAN

(11)Publication number:

62-022790

(43) Date of publication of application: 30.01.1987

(51)Int.Cl.

C07F 7/18 CO7F 7/08

(21)Application number: 60-163548

(71)Applicant: SHIN ETSU CHEM CO LTD

(22)Date of filing:

24.07.1985

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ISHIHARA TOSHINOBU

YAMAMOTO AKIRA

(54) PRODUCTION OF TERTIARY HYDROCARBONSILYL COMPOUND

(57) Abstract:

PURPOSE: To safely and readily obtain the titled compound useful as a special silylating agent used for synthesizing medicines without using dangerous Li compounds, by reacting a Grignard reagent with a specific organosilicon compound in an organic solvent.

CONSTITUTION: (A) A Grignard reagent expressed by the formula R1MgX (R1 is tertiary hydrocarbon; X is halogen), e.g. tert-butylmagnesium chloride, is reacted with (B) an organosilicon compound expressed by the formula AmR2nSiH4-m-n [R2 is (substituted) monofunctional hydrocarbon; a is alkoxy; m is 1,2 or 3; n is 0, 1 or 2; m+n≤3], e.g. dimethylmethoxysilane, in an organic solvent, preferably THF, preferably at 40W100°C in an inert atmosphere to afford the aimed compound, e.g. tert-butylethoxysilane.

LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection1

http://www19.ipdl.ncipi.go.jp/PA1/cgi-bin/PA1DETAIL

26.04.20

'JROPEAN PATENT OFF DE

Patent Abstracts of Japan

PUBLICATION NUMBER

: 62022790

PUBLICATION DATE

30-01-87

APPLICATION DATE

24-07-85

APPLICATION NUMBER

60163548

APPLICANT: SHIN ETSU CHEM CO LTD;

INVENTOR: YAMAMOTO AKIRA;

INT.CL.

: C07F 7/18 C07F 7/08

TITLE

: PRODUCTION OF TERTIARY HYDROCARBONSILYL COMPOUND

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 $A_mR^2{}_nSiH_{4-m-n}$ [R² is (substituted) monofunctional hydrocarbon; a is alkoxy; m is 1,2 or 3; n is 0, 1 or 2; m+n \leq 3], e.g. dimethylmethoxysilane, in an organic solvent, preferably THF, preferably at 40~100°C in an inert atmosphere to afford the

aimed compound, e.g. tert-butylethoxysilane.

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feed of MeSiCl3 to give Ph2SiMeCl/PhMeSiCl2 in controllable ratios.

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NOTE: continuous process, ratio of products depends on ratio of reactants

REFERENCE COUNT: 9 THERE ARE 9 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

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ACCESSION NUMBER: 138:4688 CASREACT

Method of preparing triphenylsilanol by phenylation of TITLE:

chloro(phenyl)silanes with phenylmagnesium chloride

and subsequent reaction with water in mixed

THF-toluene solvent

Zhung V. 1 Zhun, A. B.; Polivanov, A. N.; Chernyshev, E. A. INVENTOR(S):

PATENT ASSIGNEE(S): Obshchestvo s Ogranichennoi Otvetstvennost'yu NPP

"MAGNOS", Russia

Russ., No pp. given SOURCE:

CODEN: RUXXE7

DOCUMENT TYPE: Patent

LANGUAGE: Russian /

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

PATENT NO. KIND DATE APPLICATION NO. DATE ---------------RU 2174124 20010927 RU 1999-125791 19991203 PRIORITY APPLN. INFO.:

RU 1999-125791 19991203 Triphenylsilanol is prepared by reaction of a phenylchlorosilane, e.g., Ph2SiCl2 or PhSiCl3, with H2O in an organic solvent such that the phenylchlorosilane is treated with PhMgCl in a mixture of THF and toluene, and then the reaction mixture is treated with H2O in the same solvents, whereupon the desired product is isolated; the THF to toluene ratio used ranges from 1:3 to 3:1 by volume, resp. In an example, treating 500 g Ph2SiCl2 with PhMgCl generated in situ from 225 g PhCl and Mg in a mixture of 250 mL THF and 250 mL PhMe and holding the mixture at room temperature 6 h followed by treatment with H2O gave 94.2% Ph3SiOH. This method makes it possible to prepare the desired product without isolation of Ph3SiCl from the reaction mixture followed by treatment with H2O and recovery of final product and also to use a mixture of solvents, thus causing greater process selectivity.